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Performance of a quadrupole mass spectrometer with a surface ionization source

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The performance of a quadrupole mass spectrometer with a surface ionization ion source has been discussed The mass spectrometer has a resolution ~ 100 at half of the peak height for mass 71 tested so far. The performance of the modified ion source has also been discussed

1. Introduction

Since the introduction of quadrupole mass spectrometry using r f field by Paul *et al* (1953), the subject has been extensively developed and studied Dawson *et al* (1959) have reviewed the subject with an extensive list of references in their paper as regards the instrumentation and uses Dey *et al* (1973) have already reported the setting up of such a mass spectrometer In the present paper we discuss the performance of the quadrupole mass spectrometer with a modified surface ionization type ion source described earlier by Dey *et al* (1967)

2. Principle of Operation

The motion of a particle of mass M and charge e in the r f. field produced in the space between two pairs of hyperbolic surfaces raised to a steady potential U and an alternating potential $V \cos \omega t$ is described by Mathieu's differential equations.

Solutions of Mathieu's equations show that injected ions perform oscillations perpendicular to the Z axis, the direction of propagation of ions. It is found that stable solutions are obtained only for certain values of a and q *i e* U, V, r_0 and

frequency $f(=\omega/2\pi)$ held constant, for certain mass or mass range of ions, a and q are given by

$$\left.\begin{aligned} \frac{8eU}{\omega^2 M r_0^2} &= a \\ \text{and} \qquad \frac{4eV}{\omega^2 M r_0^2} &= q \end{aligned}\right\} \qquad \ldots (1)$$

where ω is the circular frequency and r_0 is the field radius, M, the mass of ion.

For maximum resolution ($R = M/\Delta M$) of the mass spectrometer, $\Delta M = 1$ and this corresponds to $U/V = 1/6$.

The maximum possible diameter of the ion entrance aperture under the condition of maximum resolution is

$$d \approx r_0/R^{1/2} \qquad \ldots (2)$$

R having been defined at 10% of the peak height.

3. Instrumentation

(a) *Mechanical* : The hyperbolic surfaces were approximated by four 200mm long, 8 mm, dia. ($= 2r$) circular, stainless steel rods machined to an accuracy of ± 0.038 mm Figure 1 shows the mounting of the rods on two machinable

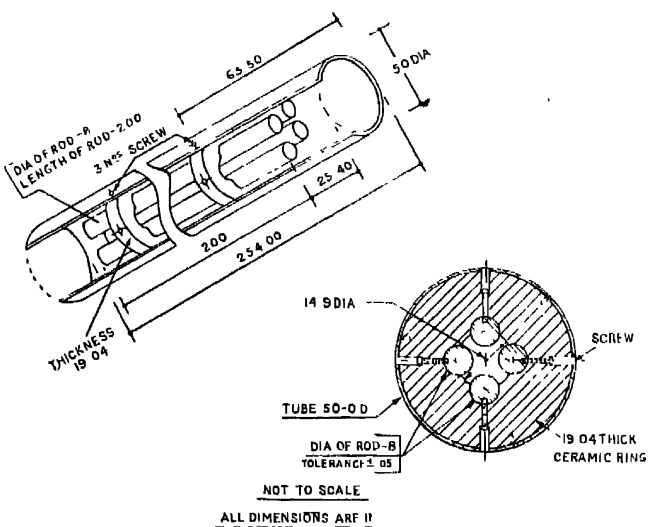

Fig. 1. Mounting of the quadrupole electrodes.

ceramics circular discs. The parameter $2(2r+r_0) = 22.9$ mm, with $2r_0 = 6.9$ mm, was maintained along the entire length of the rods to an accuracy of ± 0.038 mm. The ratio of rod diameter to field diameter was 1.16.

(b) *Vacuum Chamber* The entire mass spectrometer including the i) ion source, (ii) electron multiplier detector and (iii) electrometer were encased in a metallic chamber which was evacuated to a pressure of $\sim 10^{-5}$ mm. of Hg by oil diffusion pumps provided with liquid air traps

4 Electronics

(a) *R F Generator* The mass spectrometer was designed to attain $M/\Delta M$ -- 100 (defined at 10% of peak hight) for $M \sim 100$. A radio frequency of 4 MHz having been chosen, the r f power requirement (with $Q \approx 300$ and $C \approx 75$ pf) was 60 watts This was ensured by using a Philips double tetrode QQE/06/40 as the power amplifier (figure 2a) The design was based round this tube, which was very rugged and it withstood temporary overloads during tuning adjustments that caused the plates to glow red To achieve the desired resolution, the frequency should be stabilized to better than $0.25/R$ This was attained by having a quartz crystal controlled master oscillator operating at 2MHz As doubler, a EF80 pentode was used This was followed by a driver amplifier E81L, which developed about 5 watts and the necessary 100 volts grid drive of QQE/06/40 All coils were housed inside I F transformer cans ($1\frac{1}{2}'' \times 2'' \times 4''$high) The power amplifier coil was wound on a 2" dia threaded ergan tube, 5" in height The r. f. choke at its plate was wound on a ferrite antenna rod 360 pf air dielectric gang condensers were used in all variable condenser positions

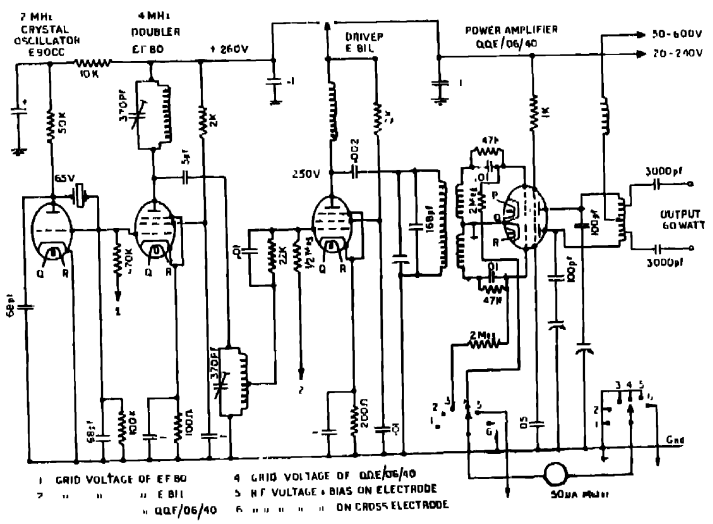

Fig. 2a R. F generator

The quadrupole rods were resonated with two coils of 25 turns each wound on double threads with 20 SWG enamelled wires, at ten turns per inch on a 2" diameter, 8" long ergan tube. R.F. heating caused very troublesome tuning

drifts at high power, with smaller coils These had an unloaded Q close to 300 and they were tapped at 7 turns for developing the positive and negative D C bias with 6AL5 rectifiers (figure 2b). The magnitude could be precisely adjusted to the desired amplitude by the $50K\ \Omega$ series potentiometers These could be measured at two positions of the six-position switch associated with the $50\mu A$ meter, which was also used for monitoring the crystal oscillator, doubler, driver and power amplifier grid biases The electrode pairs were individually tuned with 10 pf variables which took up also the unbalances. The power amplifier was tuned to the minimum plate current as the gang condenser was swung through resonance

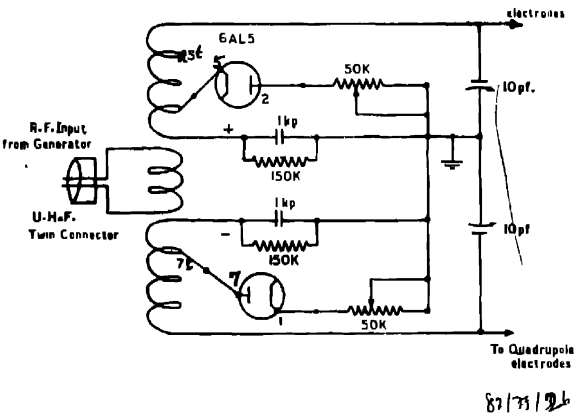

Fig 2b. Electrode biasing and tuning circuit

The high tension supply for the r f generator was stabilized to better than $\frac{1}{2}R$ of its value to attain the desired resolution of the mass spectrometer.

5. Performance

The performance of the mass spectrometer was tested using a surface ionization type source with modification reported earlier by Dey *et al* (1967). The ion source used in the present experiment was almost of the earlier type excepting that the ionizing tungsten filament was coiled instead of being straight so as to increase the area of ionization and also to avoid any misalignment of the filament with respect to the slits used for accelerating and collimating the ion beam, when it was heated The evaporating filament was surrounded by a pyrex glass capsule with a radial hole at its centre facing the ionizing filament The capsule was loaded with the charge materials and when current passed through the evaporating filament, vapour coming out of the hole directly struck the heated ionizing fialment By this technique one can do away with separate evaporators, generally used in surface ionization sources, without much loss in efficiency

The ions were accelerted through a potential difference of 150-200V with the exit slit at zero potential, the diameter of it being 0·36 mm, according to eq (2).

The ions, after transmission through the quadrupole, were detected by a 10-stage Ag-Mg electron multiplier (Dumont SP 182) operated at a gain of 10^5. The output was further amplified by an electrometer and finally recorded with an Easterline Angus 0-1mA pen recorder.

Mass scanning was carried out by varying the r f and the associated d.c. voltage in the ratio of 6·1, with a small synchronous motor

6. Results and Discussion

For the study of ionization of Potassium and Gallium, the charge materials used were KCl (Merck) and $GaF_3\,3H_2O$ (Fluka AG) The transmitted currents were 10^{-13}A for K^+ and 10^{-15}A for Ga^+ Mass analysed isotopes displayed on the pen recorder for K and Ga in one of the runs are shown in figures 3 and 4 respectively

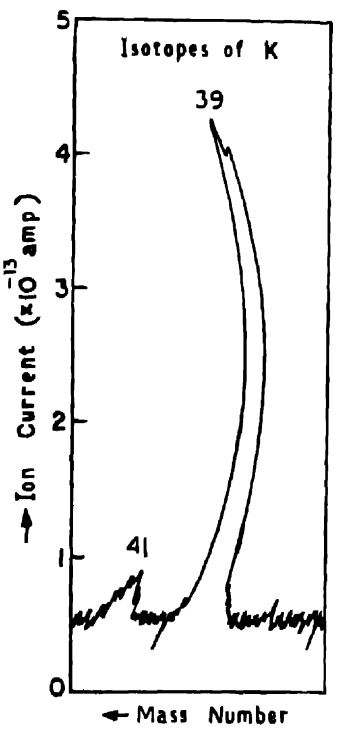

Fig. 3 Isotopes of K.

The relative isotopic abandance ratios of K and Ga were measured in six different runs from the same samples The average values were 1.37 ± 0.03

for K and 1·48±0·02 for Ga, in good agreement with the published values 1·35 and 1·50 respectively (Weast 1966-1967) Reliable measurement of isotopic abundance ratio is thus possible using the modified surface ionization ion source even when the degree of ionization α is small According to Saha-Langmuir equation, (Langmuir *et al* 1925), α is small when the ionization potential of the evaporating atoms is greater than the work function of the filament material (e g. for ionization of Ga on *W*)

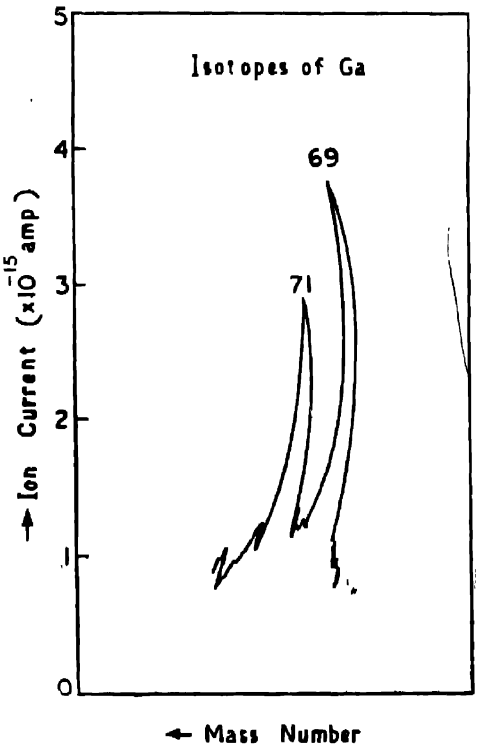

Fig. 4. Isotopes of Ga

A resolution ~ 100 was attained at half with of the peak height which is less than that anticipated by the design considerations The discrepancy is likely due to the approximation of the hyperbolic surfaces by the circular ones which produce a lower resolution as experimentally verified by Brubaker *et al* (1967, 1968) Since the field radius r_0 appear in a and q to the second power (eq (1)), the field is required to be stabilized to better than 1/4R of its value It is likely that in course of operation of the mass spectrometer, deposit of foreign materials may be responsible for altering the desired field stability causing non linear resonances as has been suggested by Levine *et al* (1965). Secondly, the mechanical

alignment of the rods over the whole field length may not be as accurate as required by the stability criterion.

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